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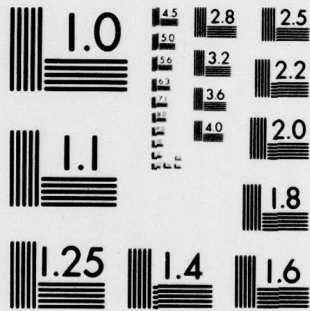
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DEPENDENCE OF THE EXTRACTION CAPACITY OF AMINES  
ON THEIR IONIZATION POTENTIALS

by

V. M. Makarov



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## EDITED TRANSLATION

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Block	Italic	Transliteration	Block	Italic	Transliteration
А а	<i>А а</i>	A, a	Р р	<i>Р р</i>	R, r
Б б	<i>Б б</i>	B, b	С с	<i>С с</i>	S, s
В в	<i>В в</i>	V, v	Т т	<i>Т т</i>	T, t
Г г	<i>Г г</i>	G, g	У у	<i>У у</i>	U, u
Д д	<i>Д д</i>	D, d	Ф ф	<i>Ф ф</i>	F, f
Е е	<i>Е е</i>	Ye, ye; E, e*	Х х	<i>Х х</i>	Kh, kh
Ж ж	<i>Ж ж</i>	Zh, zh	Ц ц	<i>Ц ц</i>	Ts, ts
З з	<i>З з</i>	Z, z	Ч ч	<i>Ч ч</i>	Ch, ch
И и	<i>И и</i>	I, i	Ш ш	<i>Ш ш</i>	Sh, sh
Й й	<i>Й й</i>	Y, y	Щ щ	<i>Щ щ</i>	Shch, shch
К к	<i>К к</i>	K, k	Ъ ъ	<i>Ъ ъ</i>	"
Л л	<i>Л л</i>	L, l	Ы ы	<i>Ы ы</i>	Y, y
М м	<i>М м</i>	M, m	Ь ь	<i>Ь ь</i>	'
Н н	<i>Н н</i>	N, n	Э э	<i>Э э</i>	E, e
О о	<i>О о</i>	O, o	Ю ю	<i>Ю ю</i>	Yu, yu
П п	<i>П п</i>	P, p	Я я	<i>Я я</i>	Ya, ya

\*ye initially, after vowels, and after ъ, ы; e elsewhere.  
When written as ё in Russian, transliterate as yë or ë.

## RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	sinh <sup>-1</sup>
cos	cos	ch	cosh	arc ch	cosh <sup>-1</sup>
tg	tan	th	tanh	arc th	tanh <sup>-1</sup>
ctg	cot	cth	coth	arc cth	coth <sup>-1</sup>
sec	sec	sch	sech	arc sch	sech <sup>-1</sup>
cosec	csc	csch	csch	arc csch	csch <sup>-1</sup>

Russian	English
rot	curl
lg	log

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## DEPENDENCE OF THE EXTRACTION CAPACITY OF AMINES ON THEIR IONIZATION POTENTIALS

V. M. Makarov

The extraction of metal salts and acids by the salts of high-molecular amines can be considered as one which occurs both by means of the ion-exchange mechanism and a combination mechanism [1-11]. Independently of the extraction mechanism, the removal of metal salts is due to the formation of a complex compound:  $[\text{Amine} \cdot \text{H}]_n \text{MA}_{m+n}$ , which, almost totally, changes into an organic phase [12-15]. In the organic phase this complex is solvated by the molecules of a solvent (Solv.), amine salt (Amine HA) and, under certain conditions, it is necessary to take into account the formation of dimers and other polyparticles. Consequently, in a general form, the composition of a complex in an organic phase can be presented by the formula

$$\{p(\text{Amine HA}) \cdot s(\text{Solv.}) \cdot [(\text{Amine H})_n \cdot (\text{MA}_{m+n})]\}$$

When investigating the effect of the composition and the amine structure on the extraction capability of its salts, it is assumed that the polymerization and solvation of the complex in an organic phase do not vary. In order to obtain quantitative regularities, Rozen and Nagnibeda used the effective extraction constants  $\bar{K}$  as a characteristic of the extraction capacity, which were calculated from the supposition that the composition of the plutonium (IV) and uranyl complexes in an organic phase corresponds to the formulas

FIRST LINE OF TEXT [Amine H]<sub>2</sub>Pu(NO<sub>3</sub>)<sub>6</sub> and [Amine H]UO<sub>2</sub>(NO<sub>3</sub>)<sub>3</sub>

and does not vary with a change in the amine composition. Taft constants  $\sigma$  and the electronegativity of the substituent group were used as a characteristic of the extractant structure [16]. Earlier, these structure characteristics were used to correlate the basicity of amines [17-19]. The authors obtained a satisfactory correlation between the extracting capacity of the amine salts and their structure during the extraction of the nitrous acid and uranyl and plutonium (IV) nitrates. The low accuracy, especially in the case of amines which have long-chain radicals, is due to the effect of the steric and entropic factors, as explained by the authors.

If the logarithms of the effective extraction constants are functions which yield to an analytical expression in a form of the Hammett equation, then they must obey the rule of polylinearity [20]. Fig. 1 shows the dependence of the logarithm of the effective extraction constant of the plutonium nitrate (IV) on the logarithm of the effective extraction constant of the uranyl nitrate into solutions of amines in CCl<sub>4</sub> according to the data of [16]. As seen from Fig. 1, there is a significant deviation of the experimental data from the linear dependence. The absence of any sort of regularity in the nature of deviations of the experimental points from a linear dependence makes it possible to assume that they are caused, apparently, either by the presence of impurities in the amines or by the insufficient accuracy of the experimental data. It is possible that this explains the relatively low accuracy in the correlation of the logarithm of the effective extraction constant with the Taft constants and the electronegativity of the substituent group [16].

The extractability of the amines is due to the fact that the nitrogen atom has an unshared pair of electrons which forms a coordination bond with the electro-acceptor atoms and ions [21-25]. In the extracting compounds the bond is formed as a result of an electron moving from the higher filled molecular orbit of a donor to a vacant molecular orbit of an acceptor; in this case, a complex is formed with a transfer of charge [26, 27]. The ground

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state 1-1 complex,  $D+A=D \cdot A=D^+A^-$ , is described by the wave function

$$\psi_R = a\psi_0(D \cdot A) + b\psi_1(D^+A^-), \quad (1)$$

where  $\psi_0$  - wave function corresponding to the van der Waals bond,  $\psi_1$  - wave function of ionic state, corresponding to a complete transfer of an electron from D to A. The square of the coefficient

$$b = \frac{cS}{I_D - E_A} \quad (2)$$

is a measure of the transfer charge. Here,  $c$  - constant,  $S$  - integral of overlapping between the donor and acceptor orbitals and it reflects the degree of superposition of both structures,  $I_D$  - donor ionization potential, and  $E_A$  - affinity of an acceptor to an electron. Since  $E_A < I_D$ , as a rule, complete ionization does not occur. From an analysis of the equation (2) we can see that the smaller the ionization potential of amine the higher the polarity of the bond and the greater is its strength.

A similar analysis carried out by Pechalin and Panchenkov [28] on the role of the donor-acceptor interaction in the processes of isotope separation by chemical exchange resulted in a satisfactory correlation between a single isotope separation coefficient and the ionization potential:

$$\alpha = m - n \cdot I_D, \quad (3)$$

where  $m$  and  $n$  are constants.

Unfortunately, the data on the ionization potentials of amines are limited and pertain to low-molecular amines which are not used as extractants. Lenard-Jones and Hall [29] have proposed an operational method for calculating the ionization potentials of the saturated hydrocarbons employing the concept of an equivalent orbital. Later this method was extended for calculating the ionization potentials of radicals [30] and other molecules [31].

According to the Lenard-Jones and Hall method, the vertical ionization potential of methane and its analogs is determined by diagonalization of the matrix of an equivalent orbital which has the form

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$$e_{ms} = \begin{vmatrix} a & b & b & b \\ b & a & b & b \\ b & b & a & b \\ b & b & b & a \end{vmatrix}.$$

The final formula for the calculation of the ionization potential has the form

$$I = -(a - 3b). \quad (4)$$

If we know the ionization potentials of several terms in a series, it is possible to determine the values of the  $a$  and  $b$  parameters in the equation (4) and calculate the ionization potential of the other terms of the same series.

From the form of the equation (4) it is evident that the values of the ionization potentials must obey the rule of polylinearity and, consequently, it is possible to assume that a linear dependence should be observed between the values of the ionization potentials of the radicals and the saturated hydrocarbons. Fig. 2 shows the dependence of the ionization potential of the saturated hydrocarbon on the sum of the ionization potentials of the radical which can compose a molecule. In this case, each saturated hydrocarbon molecule was assumed to consist of the  $-\text{CH}_2-$  group with the appropriate radicals attached to each end. The values for the ionization potential were taken from [32]. The ionization potentials of the long-chain saturated radicals were calculated using the Hall equation [33]

$$I_{\text{rad.}} = 8.565 - 0.153 \cos\left(\frac{m}{\pi} + 1\right), \quad (5)$$

where  $I_{\text{rad.}}$  - radical ionization potential in eV,  $m$  - number of the C-C bonds in the radical. The calculated values of the ionization potentials are given in Table 1.

As can be seen from the data presented in Fig. 2, a satisfactory correlation is obtained and the ionization potential of the saturated hydrocarbon can be determined using a straight-line equation:  $I = 6.01 + 0.252 \sum I_{\text{rad.}}$ . The result that was obtained made it possible to assume that, apparently, one should be able to observe a linear dependence of the ionization potential of amine on the sum of the ionization potentials of the radicals which are its

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constituents. The literature values of the ionization potentials of amines are presented in Table 2 [32].

Figure 3 shows a dependence of the ionization potential of amine on the sum of the ionization potentials of the radicals which are its constituents. The observed linear dependence of the ionization potential of amine on the sum of the ionization potentials of radicals is expressed by the straight-line equation

$$I_{\text{am.}} = 3.20 + 0.173 \sum I_{\text{rad.}} \quad (6)$$

where  $I_{\text{am.}}$  - ionization potential of amine and  $I_{\text{rad.}}$  - ionization potential of a radical. In this case, unique correlation is observed for the primary, secondary, and tertiary amines.

Table 3 shows the values of the effective constants of extraction of the nitrous acid and uranyl and plutonium (IV) nitrates according to the data of [16] and the ionization potentials of amines, calculated using equation (6).

Since the difference in the values of the ionization potentials of the radicals with a normal structure and isostructure decreases with an increase in the number of the carbon atoms, the n.-C<sub>8</sub>H<sub>17</sub> ionization potential was accepted for the iso-C<sub>8</sub>H<sub>17</sub> radical. For extractant having a cycle (position 6), the ionization potential was calculated from a comparison of the ionization potentials of cyclic amines and cyclic radicals [32] and it should comprise 7.6-7.7 eV. Logarithms of the effective extraction constants are compared with the ionization potentials on Fig. 4. We observed a linear dependence in an analytical form which is expressed by the equation

$$\lg K = A - B \cdot I_{\text{am.}} \quad (7)$$

and with the consideration of the dependence of the ionization potential of amine on the sum of the ionization potentials of the radicals by the equation

$$\lg K = A' - B' \cdot \sum I_{\text{rad.}} \quad (8)$$

Constants of equations (7) and (8) have the following values:

	A	B	A'	B'
Uranium (VI)	22.7	2.85	13.58	0.493
Plutonium (IV)	39.87	4.48	25.53	0.775
HNO <sub>3</sub>	9.26	1.28	5.17	0.222

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The rather low accuracy of correlation is explained, apparently, not only by the effect of the entropic and steric factors but also by a low purity of the amines used in the experiment.

The determined linear dependence of the logarithm of the effective constant of extraction by the amine salts on the value of the ionization potential of the amine is an empirical rule, since it does not take into account the effect of solvation of the amine and its salt, steric factors, variations in the ionization potentials of amines during the formation of salts and complexes, affinity of amine to electron which should acquire a particularly large significance in the case of amines, which have substituents with high affinity to an electron and a low ionization potential, of the phenyl type, and other reasons.

From the nature of the dependence of the logarithm of an effective extraction constant on the ionization potential (Fig. 4) it is evident that the difference in the extraction capacity of the amine salt increases with a decrease in the ionization potential of the amine relative to the uranyl and plutonium. In this case the logarithm of an effective separation coefficient determined by the ratio of the effective extraction constants has a linear dependence on the ionization potential of the amine:

$$\lg \alpha_{\text{pau}} = \lg K_{\text{pu}}/K_{\text{U}} = 17.17 - 1.63 \cdot I_{\text{au}}. \quad (9)$$

Equation (9) does not take into account the effect of steric factors on the nature of variation in the effective separation coefficient during a change in the composition and structure of the amines which, apparently, can have a higher value, under certain conditions, than the energy factors [34].

## CONCLUSIONS

1. It was shown that the ionization potentials of primary, secondary, and tertiary amines have a linear dependence on the sum of the ionization potentials of the substituents.
2. It was established that the logarithm of an effective extraction constant is a linear function of the amine's ionization potential and the logarithm of the effective coefficient of separa-

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tion of the plutonium (IV) and uranyl nitrates during their extraction by the amine salts increases with a decrease in the ionization potential of the amine.

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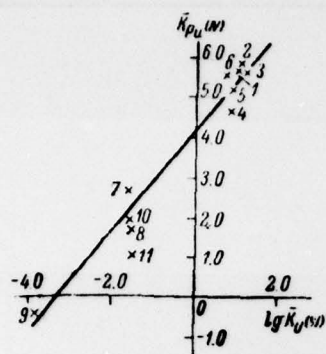


Fig. 1. Dependence of the logarithms of the effective constants of extraction of the plutonium (IV) and uranyl nitrates [16]. Numbers of points correspond to the numbers of amines in Table 3.

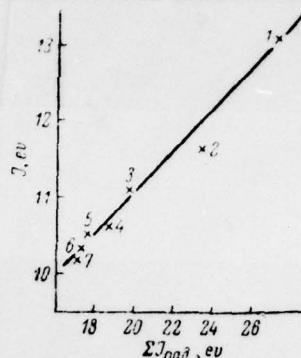


Fig. 2. Dependence of the ionization potentials of saturated hydrocarbons on the sum of the ionization potentials of radicals.

1 -  $\text{CH}_4$ , 2 -  $\text{C}_2\text{H}_6$ , 3 -  $\text{C}_3\text{H}_8$ ,  
4 -  $\text{C}_4\text{H}_{10}$ , 5 -  $\text{C}_5\text{H}_{12}$ , 6 -  $\text{C}_7\text{H}_{16}$ ,  
7 -  $\text{C}_{10}\text{H}_{22}$ .

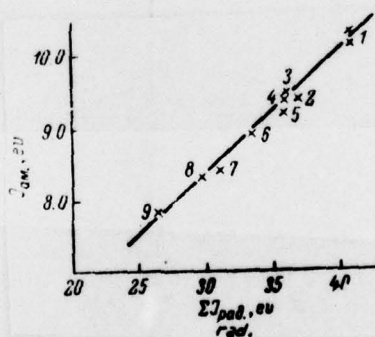


Fig. 3. Dependence of the ionization potentials of amines on the sum of the ionization potentials of radicals.

1 -  $\text{NH}_3$ , 2 -  $\text{CH}_3\text{NH}_2$ , 3 -  $\text{C}_2\text{H}_5\text{NH}_2$ , 4 -  $\text{C}_3\text{H}_7\text{NH}_2$ , 5 -  $\text{C}_4\text{H}_9\text{NH}_2$ , 6 -  $(\text{CH}_3)_2\text{NH}$ ,  
7 -  $(\text{C}_2\text{H}_5)_2\text{NH}$ , 8 -  $(\text{CH}_3)_3\text{N}$ , 9 -  $(\text{C}_2\text{H}_5)_3\text{N}$ .

TABLE 1

Values of the ionization potentials of the long-chain saturated radicals

Radical Радикал	$I$ in $I$ (eV)
$C_3H_{11}$	8.597
$C_6H_{13}$	8.573
$C_8H_{15}$	8.558
$C_8H_{17}$	8.547
$C_9H_{19}$	8.538
$C_{10}H_{21}$	8.532
$C_{12}H_{25}$	8.524

TABLE 2

Values of the ionization potentials of amines

Amine Амин	$I$ in $I$ (eV)
$NH_3$	10.34 10.15
$CH_3NH_2$	9.41
$C_2H_5NH_2$	9.5
$C_3H_7NH_2$	9.4
$C_4H_9NH_2$	9.19
$(CH_3)_2NH$	8.93
$(C_2H_5)_2NH$	8.44
$CH_3)_3N$	8.32
$(C_2H_5)_3N$	7.85

TABLE 3

Values of the effective extraction constants of uranyl, plutonium (IV), and nitrous acid; sums of the ionization potentials of radicals and ionization potentials of amines

Extractant Экстракт	$K_{HNO_3}$	$K_{U(VI)}$	$K_{Pu(IV)}$	$\Sigma I_{rad.}$ (eV) $I_{rad.}$	$I_{am.}$ (eV) $I_{am.}$
$(C_7H_{15})_3N$	0.156	10.32	$5.7 \cdot 10^5$	25.674	7.64
$(C_8H_{17})_3N$	0.263	11.71	$7.3 \cdot 10^5$	25.641	7.64
$(C_{10}H_{21})_3N$	0.318	14.30	$5.3 \cdot 10^5$	25.594	7.63
$(C_{12}H_{25})_3N$	0.425	6.63	$0.46 \cdot 10^5$	25.572	7.62
$(n\text{-}C_8H_{17})_3N$	—	7.00	$2.1 \cdot 10^5$	25.641	7.64
$CH_3(CH_2)_9NC_6H_{13}$	—	4.50	$4.22 \cdot 10^5$	—	7.6—7.7
$(C_8H_{17})_2NH$	0.023	$2.42 \cdot 10^{-3}$	491.6	30.689	8.51
$(n\text{-}C_8H_{17})_2NH$	0.027	$3.10 \cdot 10^{-3}$	53.6	30.689	8.51
$C_8H_{17}CH_2NHCH_3$	0.010	$1.54 \cdot 10^{-4}$	0.407	31.185	8.71
$C_8H_{17}CH_2NHC_6H_{13}$	—	$2.55 \cdot 10^{-3}$	82.9	29.849	8.36
$n\text{-}C_8H_{17}NH_2$	—	$3.10 \cdot 10^{-3}$	11.0	35.737	9.39



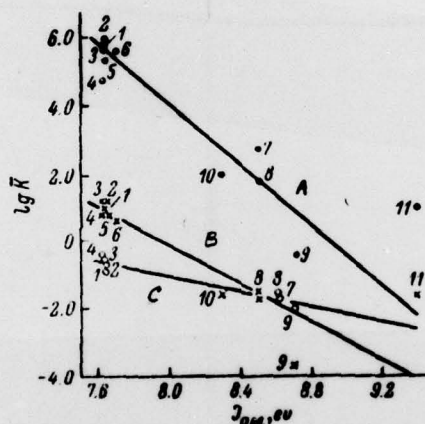


Fig. 4. Dependence of the effective extraction constants on the ionization potentials of amines. The numbers of points correspond to the numbers of the amines in Table 3.

A - nitrate  $\text{Pu}^{\text{IV}}$ , B - uranyl nitrate, C - nitrous acid.



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P005 DOE	1		
P050 CIA/CRB/ADD/SD	2		
NAVORDSTA (50L)	1		
NASA/KSI	1		
AFIT/LD	1		
ILL/Code L-389	1		
NSA/1213/TDL	2		